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Data Article

Data of OECD soil and leachate resulting from irrigation with aqueous solution containing trace metals at increasing sodium concentration



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ABSTRACT

The accumulation of trace metals in soils is one of the main drawbacks when reclaimed waters are used as irrigation sources in agriculture. Such secondary water sources often also contain high levels of salinity and in detail sodium. How the presence of sodium influences the fate of trace metals in the environment is still obscure and of primary importance in defining sustainable agricultural management guidelines. Standard OECD soil columns were subjected to 25 days irrigation with aqueous solutions containing trace concentrations of Cd, Cu, Ni and Zn at increasing sodium content. The dataset is supplementary to the data presented and discussed in "Effect of sodium concentration on mobilization and fate of trace metals in standard OECD soil" [1]. The leachates collected from the columns were deeply characterized in terms of concentration of metals, organic (monitored through UV-VIS, 3DEEM, E_4/E_6 ratio, COD) and inorganic matter

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(monitored as Al concentration). The dataset was analyzed by PCA and “Paerson” correlation coefficient.

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Specifications Table

Subject area	Chemistry, Engineering, Agronomy,
More specific subject area	Environmental Engineering
Type of data	Table, graph, 3DEEM images, graphs
How data was acquired	ICP-MS (Nexion 3000 – Perkin Elmer, USA), Spectrofluorometer (FP 750 – Jasco, Japan), UV-VIS spectrophotometer (V-530 – Jasco, Japan), UV-VIS spectrophotometer (Photolab 6600 – WTW, Germany)
Data format	Raw, analyzed,
Experimental factors	Soil samples were cut in slices and mineralized by microwave digestion in aqua regia. Leachate samples were acidified with 1% HNO_3 or analyzed raw.
Experimental features	Leaching column experiments to assess the fate of selected trace metals in standard soil
Data source location	Naples, Italy, University of Naples Federico II, 40° 49' 42.8" N 14° 11' 25.2" E
Data accessibility	Data with this article
Related research article	“Effect of sodium concentration on mobilization and fate of trace metals in standard OECD soil. Environmental Pollution, 2019. 250 : p. 839–848”. [1]

Value of the data

- Dataset reports some properties of organic matter released from soil in different sodicity conditions
- Data assess the accumulation/mobilization of trace metals (TMs) in different sodicity conditions
- OECD reference soil is widely used to mimic complex solid samples and its interactions with contaminants is of primary importance since it affects the definition of environmental risk thresholds.
- OECD soil was developed as growth medium soil to test the eco-toxicity of chemicals on selected species of earthworms. Data could be used to assess the effect of sodium on the results of OECD tests.

1. Data

OECD soil was irrigated with an artificial solution of four different TMs with the concentrations reported in [Table 1](#). The data of the leachate characterization are reported in [Table 3](#) for each of the replicates. Average standard deviation among the technical replicates of E_4/E_6 values are reported in [Table 2](#).

The dataset was analyzed by the Paerson correlation coefficients which are reported in [Table 4](#). Correlations are highlighted in color scale. Statistical analysis performed on the dataset related to 0 mM [Na] indicated a correlation ($r_{\text{TMs}} > 0.9$) among all the TMs and correlation with Al ($r_{\text{TMs-Al}} \approx 0.5$). [Na] ranging between 1 and 5 mM caused correlation among TMs change ($0.2 < r_{\text{TMs}} < 0.6$), also with released Al ($0 < r_{\text{TMs-Al}} < 0.2$ at 5 mM [Na]). Correlations with other parameters (E_4/E_6 , FI@275, FI@370, COD) also changed at increasing sodicity. PCA analysis is reported in [Fig. 2](#) at each [Na] tested.

The fluorescence matrixes of the leachates at each [Na] tested are reported in [Fig. 1](#). The relative maximum fluorescence areas detected were corresponding to excitation at 275 nm – emission at 475 nm and excitation at 370 nm - emission at 470 nm wavelengths.

Table 1

Concentration of TMs in the synthetic reclaimed wastewater and in the OECD soil.

	Cd [ppb]	Cu [ppb]	Ni [ppb]	Zn [ppb]
Synthetic reclaimed wastewater	5	100	100	500
FAO guidelines	10	200	200	2000
Kaolinite	14.2 ± 0.6	6.26 · 10 ⁴ ± 4.0 · 10 ³	748 ± 34	1.09 · 10 ⁴ ± 4.7 · 10 ²
Sphagnum Peat	97.2 ± 4.1	2.41 · 10 ³ ± 98	1.80 · 10 ³ ± 45	1.93 · 10 ⁴ ± 2.8 · 10 ²
Quartz Sand	5.03 ± 0.12	1.91 · 10 ³ ± 70	787 ± 23	8.69 · 10 ³ ± 2.8 · 10 ²

2. Experimental design, materials and methods

The micro-pollution of a standard soil model was reproduced at laboratory scale, simulating the irrigation with an artificial solution of four different TMs with the concentrations reported in [Table 1](#) at various sodicity levels (0, 1, 5, 10 and 50 mM).

The standard soil was prepared as indicated by the guidelines of the Organization for Economic Cooperation and Development (OECD) [2]. OECD soil was developed as growth medium soil to test the eco-toxicity of chemicals on selected species of earthworms. Therefore, such reference soil is widely used to mimic complex solid samples, and its interactions with contaminants is of primary importance since it affects the definition of environmental risk thresholds. OECD soil composes of: kaolinite, 20% (w/w); quartz sand, 74% (w/w); CaCO₃, 1% (w/w); sphagnum peat, 5% (w/w). Kaolinite, quartz sand and CaCO₃ were produced by Sigma-Aldrich (USA). Acid sphagnum peat was bought from a gardening store. Before use, the soil was analyzed to define the background levels of the tested TMs. Results of the analysis are reported in [Table 1](#). TMs concentrations were chosen to be well below the threshold values suggested in the wastewater quality guidelines for agricultural use by the Food and Agriculture Organization of the United Nations (FAO) [3]. In details the different synthetic reclaimed wastewater solutions [4] were obtained dissolving analytical grade CuCl₂·2H₂O (Carlo Erba Reagenti, Italy), NiCl₂·6H₂O (Sigma-Aldrich, USA), and ZnCl₂ (Applichem, Germany) and 1000 ppb Cd analytical standard (Carlo Erba Reagenti, Italy), in analytical grade HNO₃ 1% (J.T. Baker, USA) solutions. 1 g/L stock solutions, were mixed and opportunely diluted with ultrapure water (Elga, USA) to obtain the TMs concentrations indicated in [Table 1](#). Amounts of analytical grade NaClO₄ (Sigma-Aldrich, USA) were added to reach the desired sodicity. ClO₄⁻ was chosen to minimize counter-anion effects. Final pH was adjusted to 6.8, adding a few drops of KOH 0.05 M. All the experiments were completed in duplicate.

Artificial reclaimed water solutions were fed to interact with soil columns in semi saturation conditions. The soil columns were filled daily with the respective influent water and the leachate was collected the day after. The overall leaching experiments lasted for 25 days for a total of 24 leachates collected. During the first 2 days of leaching, samples were collected with 12 h interval. Then sampling frequency was 24 h. An interval of 72 h between samplings was adopted at leaching time corresponding to days 3, 8, 13, 18.

To evaluate the accumulation and the migration in the soil column at different Na content, TMs concentration was determined at each sampling time in the leachate and, at the end of the experiment, in the soil column at different depths as described elsewhere [5]. The release of organic substances into the leachates was indirectly monitored as chemical oxygen demand (COD) and Ultra-Violet (UV)

Table 2Standard deviation of E₄/E₆ at increasing [Na].

RSD E ₄ /E ₆	Na [mM]				
	0	1	5	10	50
min	0.1%	0.4%	0.4%	0.3%	0.1%
max	44.9%	79.2%	92.0%	36.3%	12.5%
avg	17.3%	21.3%	47.2%	20.4%	5.3%

Table 3

Leachate dataset. Metals and COD concentration is expressed in mg/L. Fluorescence intensity in fluorescence units (FU).

0 Na	days	Cd	Cu	Ni	Zn	Al	E4/E6	Fl@275	Fl@370	COD
column 1	1	0.102	27.1	5.43	594.3	93.3	6.9	142.2	405.4	668.7
	1.5	0.054	13.7	5.88	115.4	34.8	7.8	239.8	374.5	239.5
	2	0.028	3.6	1.61	120.5	30.2	11.5	273.2	327.3	185.3
	3	0.018	2.9	1.36	95.1	20.3	7.7	284.3	296.3	155.3
	4	0.029	3.9	1.38	139.3	23.9	7.3	249.9	310.5	221.2
	5	0.072	3.4	1.28	158.0	19.4	9.2	312.7	241.8	165.3
	6	0.053	3.2	0.92	223.8	15.5	9.2	253.6	186.8	92.8
	7	0.030	7.1	0.96	115.1	16.0	10.5	288.2	197.5	108.8
	8	0.058	15.8	3.65	123.7	45.2	9.2	259.7	171.6	105.5
	9	0.039	9.1	14.9	299.5	30.4	10.5	245.3	176.4	100.5
	10	0.011	14.4	1.57	185.1	32.9	8.5	242.7	153.4	81.3
	11	0.002	6.0	1.38	83.4	35.3	8.8	234.3	149.0	86.9
	12	0.003	10.4	3.05	177.1	73.9	8.8	218.1	131.7	91.3
	13	0.004	6.4	0.93	66.8	100.8	6.6	195.0	115.2	68.7
	14	0.041	7.0	2.15	107.0	103.5	6.5	197.6	133.7	91.4
	15	0.775	236.3	67.7	2185.2	697.6	4.6	191.2	110.7	67.3
	16	0.039	7.9	6.26	106.9	244.4	4.6	197.5	115.7	70.5
	17	0.047	7.9	2.61	81.7	308.8	4.1	175.4	100.3	40.3
	18	0.012	6.9	2.79	78.1	353.5	3.3	191.9	113.6	23.1
	19	0.020	6.6	2.53	146.6	189.4	6.2	180.5	114.7	63.1
	20	0.024	8.4	5.93	149.7	254.2	3.5	165.3	94.2	48.1
	21	0.005	7.5	3.07	113.1	305.4	3.5	161.3	90.5	46.8
22	0.008	11.2	8.57	110.8	297.7	3.4	156.8	89.3	49.6	
column 2	1	0.064	11.7	9.68	436.4	134.1	7.0	123.1	424.8	677.0
	1.5	0.054	15.4	5.14	325.0	73.3	9.2	123.7	322.4	281.2
	2	0.029	5.0	1.67	140.6	51.2	11.6	234.9	343.7	240.3
	3	0.021	3.8	1.43	124.4	32.5	1.0	260.7	311.3	168.7
	4	0.029	6.0	0.78	123.8	36.9	0.9	228.2	310.6	242.0
	5	0.019	3.3	1.45	79.8	22.4	10.3	300.1	241.5	125.3
	6	0.019	9.0	0.48	98.1	23.8	8.6	262.7	208.0	121.2
	7	0.027	8.4	0.64	55.9	21.5	9.0	228.8	161.3	113.0
	8	0.018	6.5	1.52	135.8	27.1	8.0	219.8	144.0	119.7
	9	0.031	5.6	1.57	639.3	35.8	8.3	211.2	154.2	100.5
	10	0.014	6.4	2.39	86.4	41.4	8.5	211.3	133.3	78.0
	11	0.012	11.7	1.13	138.6	38.9	8.2	225.2	135.2	83.7
	12	0.003	10.0	0.85	47.3	55.7	8.2	199.3	115.4	76.6
	13	0.031	8.0	2.05	334.3	84.8	8.4	191.8	109.5	77.0
	14	0.025	5.9	1.85	76.5	88.8	5.7	202.5	144.0	58.0
	15	0.020	5.7	5.26	108.6	179.0	3.8	186.1	104.8	69.3
	16	0.026	7.5	2.55	141.6	329.4	3.7	176.8	98.6	29.7
	17	0.017	7.4	2.55	101.6	332.7	2.9	184.1	106.2	46.3
	18	0.012	7.3	2.27	51.8	356.0	2.2	165.5	100.8	52.7
	19	0.025	11.1	3.35	129.6	357.9	3.2	175.1	110.6	68.0
	20	0.008	7.9	2.46	211.2	327.7	2.4	165.5	99.3	72.1
	21	0.013	8.7	2.54	144.4	386.9	2.3	166.3	100.6	41.2
22	0.008	8.7	1.75	139.3	368.6	2.1	153.2	90.3	43.1	
1 Na	days	Cd	Cu	Ni	Zn	Al	E4/E6	Fl@275	Fl@370	COD
column 1	1	0.120	13.1	12.28	512.1	270.5	6.2	106.4	406.6	843.7
	1.5	0.045	14.3	6.55	262.5	86.7	9.6	224.3	467.7	269.5
	2	0.039	10.1	4.29	269.1	133.7	5.6	273.2	327.3	227.8
	3	0.032	3.0	1.14	120.5	50.8	9.1	253.3	333.3	195.3
	4	0.025	2.8	1.04	190.1	51.0	8.1	231.5	348.3	265.3
	5	0.016	2.6	0.71	55.6	16.9	9.0	285.5	248.3	163.7
	6	0.019	7.6	0.61	81.6	38.1	10.4	251.7	206.8	147.8
	7	0.021	5.7	0.64	94.0	26.6	9.3	269.1	199.3	109.7
	8	0.021	3.3	2.18	88.6	23.5	9.8	239.4	176.1	128.8
	9	0.029	5.8	1.8	193.3	30.2	10.6	245.3	190.0	138.0
	10	0.005	6.2	1.45	183.3	37.7	8.8	244.8	162.9	95.5
11	0.005	27.6	8.86	145.9	60.9	6.5	238.6	160.5	105.5	

Table 3 (continued)

1 Na	days	Cd	Cu	Ni	Zn	Al	E4/E6	FI@275	FI@370	COD
	12	0.007	5.9	1.98	91.7	84.7	9.4	228.7	150.6	136.7
	13	0.008	5.4	1.54	151.1	74.9	7.2	217.7	142.5	89.7
	14	0.042	10.0	1.45	91.2	98.4	8.1	206.2	166.9	126.5
	15	0.023	5.7	2.4	123.2	145.2	6.8	213.5	135.8	75.4
	16	0.033	4.9	9.59	177.4	90.8	8.8	212.9	128.8	85.5
	17	0.091	6.9	6.53	163.3	92.1	9.1	197.5	116.6	62.0
	18	0.022	4.0	5.50	125.9	83.1	9.4	218.4	130.4	67.3
	19	0.020	4.8	2.35	185.6	113.1	8.6	192.9	137.0	112.9
	20	0.108	6.5	6.15	130.8	88.9	8.0	189.2	112.9	51.9
	21	0.062	5.3	7.13	122.7	99.2	7.9	176.9	99.6	75.6
	22	0.043	11.3	10.92	126.1	103.5	6.2	167.6	90.5	60.4
column 2	0.5	0.062	14.2	13.54	679.8	50.7	5.7	245.5	479.1	402.0
	1	0.070	13.1	6.35	273.2	177.1	6.6	84.6	372.9	#####
	1.5	0.048	16.7	6.79	286.9	64.7	9.3	185.6	429.5	367.0
	2	0.033	8.8	1.11	217.0	68.7	8.5	250.6	417.2	240.3
	3	0.030	2.9	1.01	142.1	52.5	11.8	136.6	274.9	209.5
	4	0.026	3.7	1.00	136.2	56.4	8.5	194.9	335.3	288.7
	5	0.019	3.6	1.05	164.2	37.3	9.3	287.6	264.0	176.0
	6	0.023	3.3	0.45	97.7	32.4	11.5	248.2	209.5	136.2
	7	0.023	4.6	2.92	96.5	39.0	8.0	242.5	195.6	137.2
	8	0.020	3.5	0.88	641.3	57.1	8.6	236.0	169.1	113.0
	9	0.029	4.5	1.13	189.5	26.6	8.5	222.7	178.9	133.8
	10	0.000	3.8	1.03	99.9	17.9	8.3	228.8	154.4	93.0
	11	0.009	7.5	1.85	125.6	24.7	1.8	157.2	118.8	93.4
	12	0.084	7.6	10.56	78.7	273.1	8.1	221.7	138.1	150.1
	13	0.005	4.7	1.69	87.9	37.2	2.8	211.4	131.7	86.5
	14	0.023	5.3	1.80	155.2	59.4	8.8	235.6	182.4	127.1
	15	0.014	3.2	2.41	89.5	49.8	10.5	209.6	125.8	91.9
	16	0.010	3.7	1.83	86.0	68.3	6.0	197.6	116.3	81.6
	17	0.017	4.7	2.32	148.1	93.3	9.0	198.7	115.4	68.7
	18	0.020	4.1	7.74	74.2	88.3	5.6	183.1	105.5	70.7
	19	0.025	5.3	4.41	252.8	99.5	7.6	215.1	149.2	109.7
	20	0.066	6.4	7.29	261.1	89.2	6.5	181.0	107.5	95.6
	21	0.116	7.2	8.57	155.4	101.3	5.6	177.2	98.7	72.3
	22	0.043	5.5	4.54	93.7	117.6	4.8	169.0	95.6	73.1
5 Na	days	Cd	Cu	Ni	Zn	Al	E4/E6	FI @275	FI @370	COD
column 1	1.5	0.056	23.3	24.96	193.4	71.2	12.7	130.9	378.7	502.0
	2	0.048	7.8	10.41	135.6	93.1	9.6	157.5	348.2	319.5
	3	0.034	4.7	5.39	113.3	87.7	9.1	188.2	322.5	262.8
	4	0.024	3.3	3.21	116.3	78.1	7.5	163.8	298.7	321.2
	5	0.019	3.2	5.26	44.4	43.8	4.7	238.3	244.7	150.3
	6	0.016	7.8	1.35	62.1	46.9	8.4	231.7	208.9	143.7
	7	0.072	5.1	1.47	89.1	37.6	8.9	239.8	192.3	243.8
	8	0.021	4.1	1.12	58.5	33.0	9.0	235.0	174.8	132.2
	9	0.031	6.8	27.56	1143.0	66.2	7.8	213.3	180.4	125.5
	10	0.006	7.2	103	146.1	27.1	8.1	220.2	150.7	127.2
	11	0.020	8.5	7.12	76.8	35.4	8.7	212.7	145.3	108.1
	12	0.000	10.0	6.93	46.6	46.6	9.4	207.8	132.4	89.5
	13	0.004	6.4	6.26	36.6	47.2	1.9	220.6	139.6	91.1
	14	0.035	9.8	49.83	59.0	111.1	10.5	188.4	154.5	127.9
	15	0.015	6.7	20.05	91.1	210.9	4.5	197.7	124.5	88.9
	16	0.070	7.8	123	133.2	306.7	4.3	190.6	113.7	75.4
	17	0.018	8.4	9.55	78.5	464.2	3.4	181.0	113.8	61.6
	18	0.019	8.3	11.76	124.2	339.7	2.6	185.5	114.5	54.3
	19	0.028	11.1	25.27	184.5	366.1	3.5	171.5	123.7	118.6
	20	0.005	9.3	6.90	124.3	339.4	2.9	172.8	111.7	84.0
	21	0.011	9.0	5.38	97.3	334.8	2.8	191.3	119.7	66.7
	22	0.028	10.8	4.70	172.3	311.1	2.6	162.3	104.8	76.6
column 2	0.5	0.091	17.5	29.06	997.4	58.9	4.5	250.0	568.9	502.0

(continued on next page)

Table 3 (continued)

5 Na	days	Cd	Cu	Ni	Zn	Al	E4/E6	Fl @275	Fl @370	COD
	1	0.084	11.9	7.44	545.7	185.8	0.7	85.5	369.8	886.2
	1.5	0.008	15.2	11.88	36.1	43	7.8	143.3	377.5	371.2
	2	0.042	6.7	1.42	149.5	87.7	7.9	219.2	379.1	282.0
	3	0.038	3.3	1.56	107.6	69.3	8.2	220.0	347.5	227.8
	4	0.027	3.6	1.13	148.1	54.0	8.2	174.6	300.9	282.8
	5	0.042	3.5	3.28	123.5	48.0	9.2	247.4	238.3	155.3
	6	0.018	7.5	0.56	69.9	26.7	11.0	233.5	202.3	136.2
	7	0.024	3.9	0.56	63.5	22.7	8.1	227.1	185.2	131.3
	8	0.026	3.8	1.35	296.0	26.4	7.0	218.3	158.9	114.7
	9	0.035	6.5	1.49	237.5	31.4	7.6	197.6	172.5	136.3
	10	0.014	5.4	2.78	372.8	24.4	8.2	214.1	149.2	93.0
	11	0.009	7.7	2.26	134.3	22.6	3.6	147.5	117.9	106.9
	12	0.005	5.8	1.57	111.6	31.4	2.0	204.8	137.7	95.8
	13	0.002	5.5	1.41	63.8	26.0	8.9	198.6	132.4	91.5
	14	0.025	7.5	5.47	121.7	35.1	7.9	201.5	164.7	140.5
	15	0.009	3.4	1.58	60.9	38.2	11.1	201.6	125.1	88.1
	16	0.011	4.2	1.58	107.3	52.5	11.2	186.8	114.6	86.7
	17	0.064	3.8	3.41	155.7	62.1	9.8	184.8	114.1	72.1
	18	0.042	6.2	8.15	146.4	79.0	7.4	175.6	106.4	65.7
	19	0.099	11.8	17.48	299.8	117.9	8.7	184.0	135.1	102.4
	20	0.013	4.6	3.45	135.3	86.3	7.3	178.3	108.7	99.8
	21	0.038	6.0	4.79	93.7	108.0	8.8	167.0	100.2	52.7
	22	0.020	5.6	2.96	98.8	139.3	5.1	175.0	101.4	70.7
10 Na	days	Cd	Cu	Ni	Zn	Al	E4/E6	Fl@275	Fl@370	COD
column 1	0.5	0.072	13.6	17.75	393.2	36.0	9.1	236.8	468.5	434.5
	1.5	0.041	14.7	4.40	237.4	38.7	9.0	124.1	374.3	478.7
	2	0.078	4.6	1.31	207.3	67.3	9.3	175.4	344.1	309.5
	3	0.027	3.0	0.94	119.5	109.1	9.0	161.7	289.9	257.8
	4	0.026	3.3	1.56	184.6	90.7	7.6	140.0	275.1	353.7
	5	0.026	6.0	0.99	139.6	59.5	9.4	212.8	239.6	180.3
	6	0.020	5.9	1.29	99.4	43.3	8.8	216.1	205.3	147.8
	7	0.069	6.8	1.02	80.8	73.5	8.3	218.5	196.0	170.5
	8	0.033	6.1	1.48	127.1	46.7	7.7	218.3	173.5	157.2
	9	0.034	11.2	1.7	174.8	41.6	8.2	182.8	173.8	153.8
	10	0.010	7.1	1.51	149.2	28.7	7.9	211.9	158.5	98.8
	11	0.002	8.5	1.04	124.0	34.6	7.1	201.4	149.9	122.9
	12	0.000	9.3	1.28	86.9	38.1	7.3	200.0	140.6	104.4
	13	0.001	6.0	1.18	79.0	51.1	7.4	209.4	144.8	100.8
	14	0.026	7.9	1.53	96.5	100.4	7.2	174.7	162.9	143.7
	15	0.017	4.4	2.7	76.2	93.3	7.5	192.9	126.4	81.8
	16	0.018	6.2	3.29	134.7	145.3	8.2	190.8	122.0	84.6
	17	0.024	6.5	3.40	95.1	173.7	7.4	181.9	115.8	68.5
	18	0.012	4.9	4.31	67.0	158.5	6.5	184.5	116.1	60.4
	19	0.047	11.6	5.61	185.5	360.4	6.5	164.3	131.5	137.2
	20	0.085	9.4	8.97	168.7	176.5	6.4	177.8	114.1	88.3
	21	0.289	18.4	17.89	449.7	142.4	4.9	222.6	134.9	137.8
	22	0.262	21.2	21.40	308.7	151.7	3.9	195.4	134.9	156.2
column 2	0.5	0.077	18.3	13.57	577.6	94.0	5.7	230.7	569.0	512.0
	1	0.054	11.8	4.91	322.8	143.7	7.1	101.0	442.9	862.8
	1.5	0.041	16.7	4.53	239.1	69.8	9.2	155.6	386.7	387.0
	2	0.035	6.5	1.58	146.6	82.0	10.3	201.0	359.5	279.5
	3	0.031	3.0	2.65	121.2	76.0	9.1	216.8	342.0	228.7
	4	0.050	3.1	1.22	146.5	70.1	8.6	168.0	277.7	307.0
	5	0.019	2.5	0.55	132.4	52.7	8.9	219.6	226.0	192.0
	6	0.051	3.7	0.52	71.0	46.4	8.5	217.9	194.9	149.5
	7	0.025	6.0	1.52	128.1	52.7	9.0	213.3	179.5	154.7
	8	0.022	5.4	2.39	311.3	54.0	9.4	211.4	159.9	143.0
	9	0.021	7.7	1.37	177.1	188.8	6.4	186.4	164.3	133.8
	10	0.000	6.6	1.38	54.8	179.5	4.7	191.9	146.7	95.5
	11	0.006	7.4	3.37	94.8	155.1	7.1	144.9	121.5	119.3

Table 3 (continued)

10 Na	days	Cd	Cu	Ni	Zn	Al	E4/E6	FI@275	FI@370	COD
	12	0.002	8.2	1.21	57.3	222.2	5.5	195.6	135.2	92.9
	13	0.005	8.8	1.82	56.8	190.2	4.6	190.1	131.4	101.2
	14	0.055	19.1	53.28	95.4	310.2	5.3	211.9	158.5	143.5
	15	0.061	10.8	4.06	96.2	422.7	4.4	186.1	120.3	30.3
	16	0.013	7.0	2.16	114.8	232.3	5.2	179.9	113.5	83.8
	17	0.015	7.0	2.26	98.7	255.7	5.2	173.7	108.5	73.7
	18	0.014	6.7	5.29	101.2	195.3	5.0	174.9	107.0	57.8
	19	0.039	13.2	8.70	76.2	301.6	5.0	169.1	133.4	124.8
	20	0.107	10.2	6.34	80.8	178.3	4.1	164.3	105.6	113.7
	21	0.026	9.2	3.22	90.7	244.1	3.7	160.3	98.4	73.3
	22	0.000	8.9	1.66	169.7	296.7	3.1	173.3	109.3	191.9
50Na	days	Cd	Cu	Ni	Zn	Al	E4/E6	FI (5)275	FI (5)370	COD
column 1	05	0.105	16.4	43.02	893.8	74.7	4.3	256.0	493.4	402.0
	15	0.024	14.9	5.99	119.7	119.1	9.0	103.7	309.9	462.8
	2	0.033	5.8	4.15	61.9	99.8	9.6	120.0	267.8	332.8
	3	0.016	3.2	3.17	62.6	113.5	105	123.2	232.5	305.3
	4	0.018	5.3	2.01	62.5	149.5	7.9	83.9	185.2	407.8
	5	0.018	4.2	0.88	65.3	99.1	8.0	147.9	184.7	875.3
	6	0.057	6.6	1.19	83.4	76.9	8.1	146.8	164.0	190.3
	7	0.016	7.7	0.78	40.0	71.8	8.1	148.5	152.5	183.0
	8	0.015	7.0	0.72	33.3	595	7.8	155.2	144.4	172.2
	9	0.025	11.1	1.4	98.7	84.6	8.0	109.5	130.3	187.2
	10	0.012	7.6	2.32	190.1	52.4	7.8	150.3	131.3	135.5
	11	0.000	11.3	1.23	62.9	54.4	7.1	142.3	123.8	127.9
	12	0.000	11.8	1.49	50.5	62.5	7.8	142.0	119.9	129.9
	13	0.000	8.5	1.02	24.5	60.9	7.6	144.1	118.6	132.7
	14	0.037	15.0	2.44	29.3	133.4	7.4	87.2	110.9	250.6
	15	0.011	7.7	1.8	31.4	117.6	7.6	146.0	112.0	104.2
	16	0.017	8.6	2.88	90.4	119.8	7.1	138.9	104.1	97.4
	17	0.016	9.6	2.18	47.6	148.7	6.2	152.6	112.9	83.0
	18	0.086	12.6	6.49	70.5	138.6	4.4	129.0	97.6	82.0
	19	0.214	27.1	14.14	101.6	226.1	4.7	95.1	100.4	265.5
	20	0.153	22.3	10.83	172.9	162.7	4.5	129.6	99.3	117.8
	21	0.118	13.7	6.69	104.6	163.5	4.6	134.5	90.4	87.1
22	0.160	18.9	12.69	88.7	140.2	3.9	127.3	88.7	101.4	
column 2	1	0.058	9.4	6.36	235.9	237.1	8.0	101.0	442.9	760.3
	15	0.040	20.4	4.37	127.4	149.4	8.4	155.6	386.7	312.8
	2	0.019	4.9	0.66	86.7	95.3	8.5	201.0	359.5	277.0
	3	0.013	3.5	0.54	70.8	832	8.5	216.8	342.0	242.0
	4	0.012	6.7	1.31	75.0	128.9	8.2	168.0	277.7	357.0
	5	0.014	4.6	0.58	47.4	61.1	8.2	219.6	226.0	183.7
	6	0.021	5.0	1.02	29.6	50.3	8.0	217.9	194.9	142.8
	7	0.018	6.0	0.35	25.0	57.8	8.4	213.3	1795	173.0
	8	0.014	7.8	1.30	77.0	445	7.2	211.4	159.9	158.0
	9	0.022	14.1	1.54	123.8	73.1	8.0	186.4	164.3	185.5
	10	0.000	9.4	4.92	40.5	39.7	7.3	191.9	146.7	113.0
	11	0.024	8.6	1.65	246.7	55.3	7.2	144.9	1215	127.9
	12	0.000	9.7	1.37	47.4	51.6	7.5	195.6	1352	109.7
	13	0.006	10.3	2.25	33.5	53.9	6.9	190.1	131.4	100.6
	14	0.021	18.1	3.78	41.8	133.8	7.1	211.9	158.5	200.1
	15	0.015	8.7	1.88	37.3	113.3	6.8	186.1	120.3	93.3
	16	0.020	10.8	2.13	41.2	147.2	6.2	179.9	113.5	103.6
	17	0.011	11.2	1.96	52.1	204.5	5.7	173.7	108.5	82.8
	18	0.021	12.8	3.71	265.3	211.6	5.1	174.9	107.0	69.7
	19	0.042	22.9	8.89	58.0	214.5	5.7	169.1	133.4	148.7
	20	0.009	12.2	3.48	39.3	165.1	4.9	164.3	105.6	114.1
	21	0.008	10.7	2.80	65.3	179.6	4.6	160.3	98.4	71.3
22	0.021	11.8	4.04	74.3	187.9	4.4	173.3	109.3	93.5	

Table 4

Paerson correlation matrix of the dataset.

0 Na	Cd	Cu	Ni	Zn	Al	E4/E6	FI@275	FI@370	COD
Cd	1.00	0.99	0.96	0.95	0.49	-0.04	-0.06	0.00	0.06
Cu	0.99	1.00	0.97	0.94	0.54	-0.09	-0.12	-0.08	-0.01
Ni	0.96	0.97	1.00	0.92	0.56	-0.10	-0.15	-0.07	0.01
Zn	0.95	0.94	0.92	1.00	0.45	-0.02	-0.16	0.04	0.17
Al	0.49	0.54	0.56	0.45	1.00	-0.69	-0.60	-0.51	-0.30
E4/E6	-0.04	-0.09	-0.10	-0.02	-0.69	1.00	0.55	0.37	0.22
FI@275	-0.06	-0.12	-0.15	-0.16	-0.60	0.55	1.00	0.27	-0.13
FI@370	0.00	-0.08	-0.07	0.04	-0.51	0.37	0.27	1.00	0.84
COD	0.06	-0.01	0.01	0.17	-0.30	0.22	-0.13	0.84	1.00
1 Na	Cd	Cu	Ni	Zn	Al	E4/E6	FI@275	FI@370	COD
Cd	1.00	0.26	0.66	0.32	0.63	-0.14	-0.47	0.16	0.39
Cu	0.26	1.00	0.57	0.33	0.28	-0.26	-0.18	0.36	0.39
Ni	0.66	0.57	1.00	0.39	0.59	-0.35	-0.41	0.09	0.29
Zn	0.32	0.33	0.39	1.00	0.21	-0.13	-0.11	0.52	0.49
Al	0.63	0.28	0.59	0.21	1.00	-0.24	-0.51	0.06	0.45
E4/E6	-0.14	-0.26	-0.35	-0.13	-0.24	1.00	0.34	0.15	-0.07
FI@275	-0.47	-0.18	-0.41	-0.11	-0.51	0.34	1.00	0.07	-0.45
FI@370	0.16	0.36	0.09	0.52	0.06	0.15	0.07	1.00	0.71
COD	0.39	0.39	0.29	0.49	0.45	-0.07	-0.45	0.71	1.00
5 Na	Cd	Cu	Ni	Zn	Al	E4/E6	FI@275	FI@370	COD
Cd	1.00	0.33	0.31	0.45	0.04	0.07	-0.13	0.43	0.51
Cu	0.33	1.00	0.58	0.30	0.23	-0.09	-0.39	0.38	0.47
Ni	0.31	0.58	1.00	0.40	0.24	0.00	-0.14	0.16	0.16
Zn	0.45	0.30	0.40	1.00	-0.04	-0.14	0.03	0.37	0.37
Al	0.04	0.23	0.24	-0.04	1.00	-0.59	-0.33	-0.27	-0.13
E4/E6	0.07	-0.09	0.00	-0.14	-0.59	1.00	0.21	0.13	-0.03
FI@275	-0.13	-0.39	-0.14	0.03	-0.33	0.21	1.00	-0.03	-0.41
FI@370	0.43	0.38	0.16	0.37	-0.27	0.13	-0.03	1.00	0.82
COD	0.51	0.47	0.16	0.37	-0.13	-0.03	-0.41	0.82	1.00
10 Na	Cd	Cu	Ni	Zn	Al	E4/E6	FI@275	FI@370	COD
Cd	1	0.605	0.466	0.565	0.039	-0.224	0.132	0.079	0.123
Cu	0.605	1	0.694	0.570	0.272	-0.405	-0.030	0.180	0.250
Ni	0.466	0.694	1	0.308	0.294	-0.287	0.182	0.064	0.075
Zn	0.565	0.570	0.308	1	-0.199	0.068	0.105	0.626	0.612
Al	0.039	0.272	0.294	-0.199	1	-0.746	-0.288	-0.438	-0.306
E4/E6	-0.224	-0.405	-0.287	0.068	-0.746	1	0.164	0.506	0.333
FI@275	0.132	-0.030	0.182	0.105	-0.288	0.164	1	-0.025	-0.350
FI@370	0.079	0.180	0.064	0.626	-0.438	0.506	-0.025	1	0.874
COD	0.123	0.250	0.075	0.612	-0.306	0.333	-0.350	0.874	1
50 Na	Cd	Cu	Ni	Zn	Al	E4/E6	FI@275	FI@370	COD
Cd	1	0.662	0.604	0.327	0.421	-0.545	-0.293	-0.009	0.062
Cu	0.662	1	0.507	0.231	0.514	-0.604	-0.195	-0.145	-0.143
Ni	0.604	0.507	1	0.853	0.184	-0.492	0.143	0.380	0.160
Zn	0.327	0.231	0.853	1	0.028	-0.284	0.234	0.522	0.243
Al	0.421	0.514	0.184	0.028	1	-0.505	-0.380	-0.054	0.116
E4/E6	-0.545	-0.604	-0.492	-0.284	-0.505	1	-0.051	0.395	0.388
FI@275	-0.293	-0.195	0.143	0.234	-0.380	-0.051	1	0.228	-0.234
FI@370	-0.009	-0.145	0.380	0.522	-0.054	0.395	0.228	1	0.645
COD	0.062	-0.143	0.160	0.243	0.116	0.388	-0.234	0.645	1

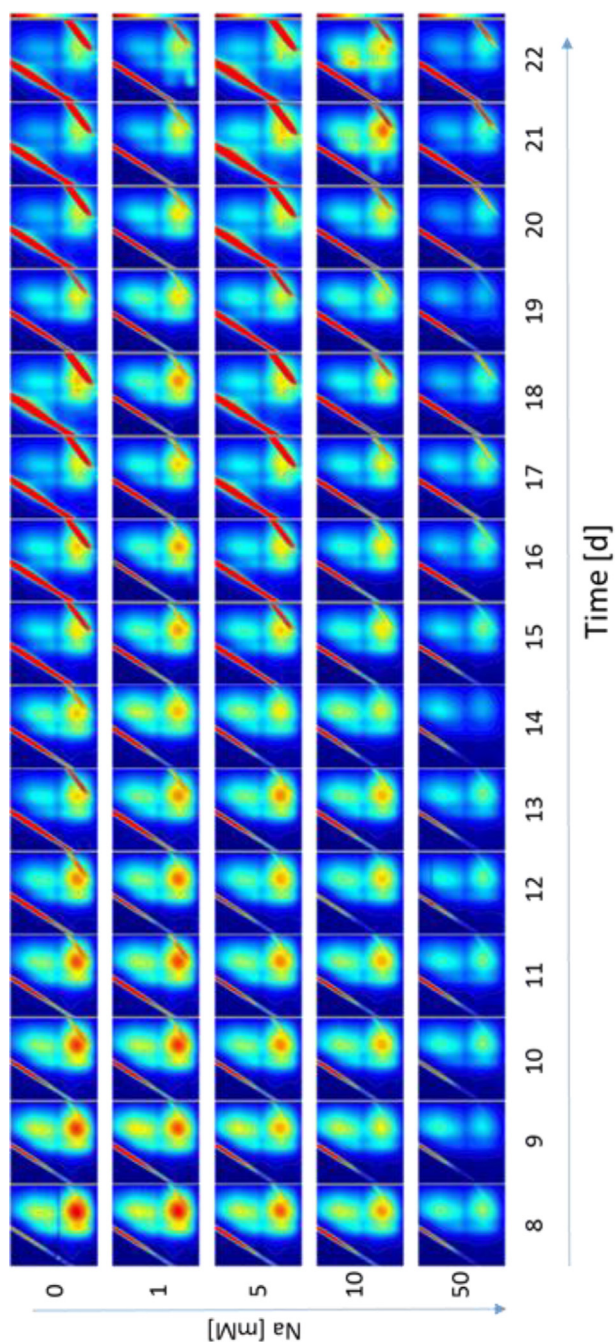


Fig. 1. 3DEEM of the leachates collected between day 8 and 22. Excitation wavelength (y axis) was varied between 220 and 450 nm; emission wavelength (x axis) was recorded between 370 and 565 nm.

absorbance. The organic matter recovered in the leachates was further characterized through three-dimensional excitation-emission matrix (3DEEM) spectrofluorometry.

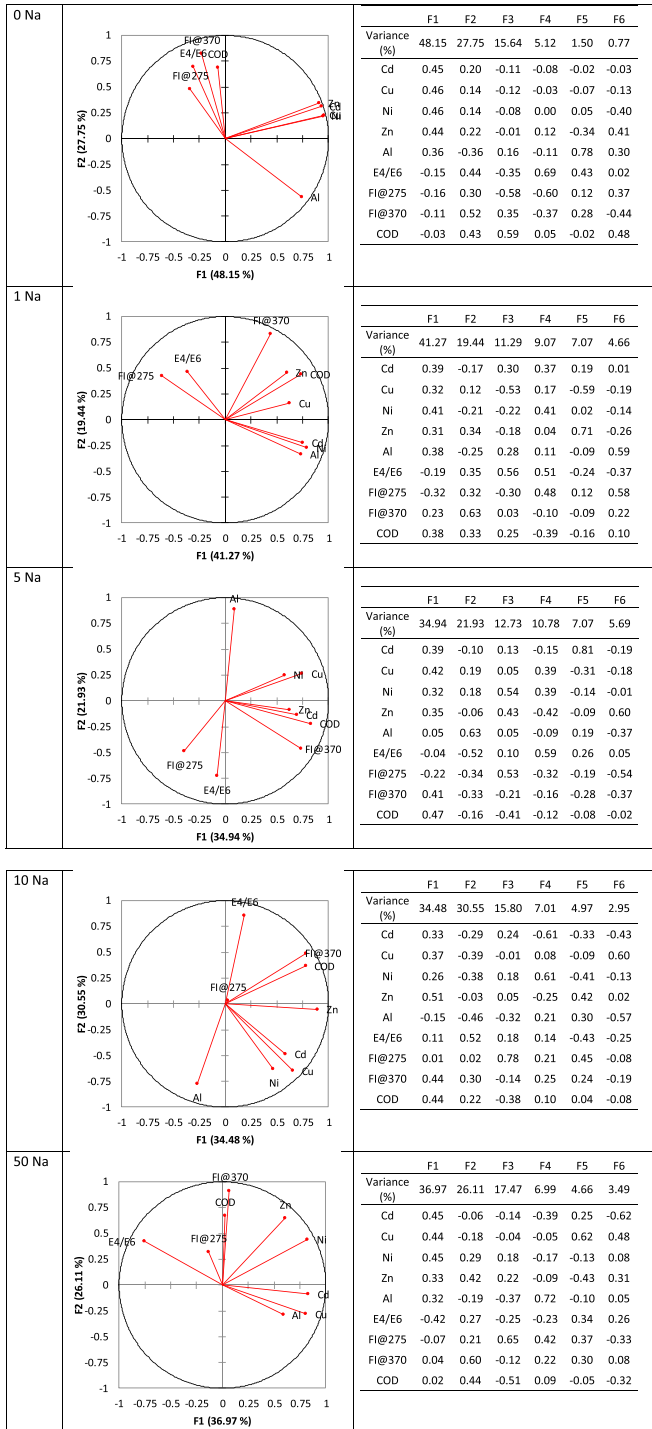


Fig. 2. PCA analysis of the dataset. (FI @275 and FI @370: fluorescence intensities measured at 275 and 370 nm excitation wavelengths respectively).

Each soil layer and the “blank” soil components were dried at 35 °C until constant weight and homogenized [6]. Aliquots of 1 g were mineralized in 15 mL of aqua regia [7] using a Milestone (USA) Start D microwave digester. Leachates were collected daily and divided into two aliquots. No filtration procedure was applied to avoid the removal of analytes bound to colloidal phases [8]. The first aliquot was acidified with 3% HNO₃ for TMs analysis. The concentrations of Al, Cd, Cu, Ni, and Zn were measured by ICP-MS (Perkin Elmer Nexion 300, USA) operating in dual detector mode. The ICP was calibrated by means of Certipur[®] certified standard multi-element solution IV (Meck – Millipore, Germany). The second aliquot was stored at –20 °C for spectroscopic characterization. Once defrosted, the UV-VIS absorbance at 245, 285, 445, 645 nm and the COD were determined. The ratio between absorbance read at 445 and 645 nm (E_4/E_6 ratio) was calculated according to Chen et al. [9]. Moreover, 3DEEM of the daily leachates was recorded. The UV-VIS measurements were carried out using a V-530 UV-VIS spectrophotometer from Jasco (Japan). COD was determined according to APHA [10] standard method 5220D. COD colorimetric measurements were acquired through a Photolab 6600 UV-VIS spectrophotometer (WTW, Germany). The 3DEEM matrices were recorded by means of the spectrofluorometer Jasco FP 750 (Japan). Emission spectra were elaborated to construct the 3DEEM through the software SPEKWIN 32. Excitation wavelength was varied between 220 and 450 nm; emission wavelength between 370 and 585 nm.

Multivariate analysis of variance was applied (MANOVA) to check for possible differences among the datasets obtained at different Na concentration. Principal component analysis (PCA) and “Pearson” correlation analysis were conducted on all the leachates datasets to evaluate the correlation structure among the parameters measured over time. Number of PCA main components was chosen in order to explain at least 90% of variance according to Pontoni et al. [1]. Statistical analyses were conducted in Microsoft[®] Excel 2013/XLSTAT[®]-Pro (Version 7.2, 2003, Addinsoft, Inc., Brooklyn, NY, USA).

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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